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<div> Division of Forensic Science CONTROLLED SUBSTANCES TRAINING MANUAL </div>	Amendment Designator: A
	Effective Date: 15-June-2005
<div> <p style="text-align: center;">16 INFRARED SPECTROPHOTOMETRY</p> <p>16.1 Objectives</p> <p>16.1.1 To familiarize the trainee with the theory and application of infrared spectrophotometry in drug analysis</p> <p>16.1.2 To familiarize the trainee with the FTIR instrumentation and software used in the laboratory</p> <p>16.2 Modes of Instruction</p> <p>16.2.1 Self-directed study through reading assignments and worksheets</p> <p>16.2.2 Presentations and demonstrations</p> <p>16.2.3 Practical exercise</p> <p>16.3 Reference</p> <p>16.3.1 Moffat, A. C., <i>et al.</i>, editors. <i>Clarke's Analysis of Drugs and Poisons</i>. London: The Pharmaceutical Press, 2004, pp. 328-345.</p> <p>16.3.2 <i>Basic Training Program for Forensic Chemists</i>, U.S. Department of Justice, Drug Enforcement Administration, Office of Science and Technology, pp. 5-17 through 5-29.</p> <p>16.3.3 DFS Controlled Substances Procedures Manual, FTIR Section.</p> <p>16.3.4 Suzuki, Edward M., Ph.D. "Forensic Applications of Infrared Spectroscopy", in Saferstein, Richard, Ph.D., editor. <i>Forensic Science Handbook, Volume III</i>. Englewood Cliffs, N. J.: Regents/Prentice Hall, 1993, pp. 71-195.</p> <p>16.3.5 Cooper, James. <i>Spectroscopic Techniques for Organic Chemists</i>. New York: John Wiley & Sons, 1980, pp. 1-52.</p> <p>16.3.6 Smith, A. Lee. <i>Applied Infrared Spectroscopy</i>. New York: John Wiley & Sons, 1979.</p> <p>16.3.7 Mills, Terry, III and Roberson, J. Conrad. <i>Instrumental Data for Drug Analysis, Second Edition</i>. New York: Elsevier, 1987.</p> <p>16.3.8 Computer-based Georgia Bureau of Investigation (Mills) library of drug compounds.</p> <p>16.3.9 Silverstein, R. M. et al. <i>Spectrometric Identification of Organic Compounds</i>. New York: John Wiley & Sons, 1991.</p> <p>16.3.10 Thermo Nicolet Instrument Manuals.</p> <p>16.4 Assignments</p> <p>16.4.1 Completion of required reading assignments (16.3.1, 16.3.3)</p> <p>16.4.2 Study questions</p> <p>16.4.3 Practical exercises</p> </div>	

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<p>16.5 Study Questions</p> <p>16.5.1 What is infrared spectrophotometry? Describe the theory behind its use as an identification technique including types of information obtained and specificity.</p> <p>16.5.2 Draw a schematic diagram for a double-beam IR.</p> <p>16.5.3 Describe the electromagnetic spectrum.</p> <p>16.5.3.1 What is the upper and lower limit on the infrared region of the electromagnetic spectrum?</p> <p>16.5.3.2 What region is the most useful analytically?</p> <p>16.5.3.3 What is the standard range of most instruments?</p> <p>16.5.4 Define the following terms:</p> <ul style="list-style-type: none"> • Wave • Wavelength • Wavenumber • Frequency • Dipole moment • Absorption • Transmittance • Overtone • Harmonic vibration • Combination band • Fundamental vibration • Monochromator • Interferometer • Homonuclear • Amplitude • Centerburst <p>16.5.5 Draw a block diagram of the FTIR and describe the function of the major components.</p> <p>16.5.5.1 Describe the different types of radiation sources for FTIR instruments.</p> <p>16.5.5.2 Describe the different types of detectors available for FTIR instruments.</p> <p>16.5.5.3 Sketch a Michaelson interferometer and describe how it works.</p> <p>16.5.6 What is “Fourier Transform” and how does it apply to IR?</p> <p>16.5.7 Explain the theory behind the Attenuated Total Reflectance (ATR) sampling unit including the differences between single-bounce and multi-bounce units.</p> <p>16.5.7.1 Describe any differences in the spectra obtained using ATR vs. regular transmittance.</p> <p>16.5.7.2 Explain the function of the ATR correction within the software including when it is permissible to use a corrected spectra in case work.</p> <p>16.5.8 What is meant by the “fingerprint region” of an IR spectrum? Why is it significant?</p>	

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<p>16.5.9 Can IR differentiate optical isomers? Diastereomers? Structural isomers?</p> <p>16.5.10 Why is polystyrene used to check the function of the FTIR?</p> <p>16.5.11 Which organic functional groups correspond to the following absorption frequencies?</p> <ul style="list-style-type: none"> • 3639-3633 cm^{-1} • 2990-2850 cm^{-1} • 1650-1510 cm^{-1} • 1750-1740 cm^{-1} • 770-690 cm^{-1} • 760-540 cm^{-1} <p>16.5.12 Why is KBr used in the preparation of solid samples?</p> <p>16.5.13 What two conditions must be met in order for infrared absorption to occur?</p> <p>16.5.14 What is the intensity of an IR absorption proportional to?</p> <p>16.5.15 Explain Beer's Law.</p> <p>16.5.16 What are the two basic categories of molecular vibration?</p> <p>16.5.17 What are the four types of bending?</p> <p>16.5.18 What is meant by vibrational coupling?</p> <p>16.5.19 Describe the differences between dispersive and non-dispersive instruments.</p> <p>16.5.20 What are the advantages of FTIR over dispersive IR?</p> <p>16.5.21 Which will vibrate with higher frequency, C-H bond or a C-C bond and why?</p> <p>16.5.22 What does hydrogen bonding do to the vibrational frequency of a hydroxyl or an amine group?</p> <p>16.5.23 Describe the absorptions for the following groups:</p> <ul style="list-style-type: none"> • -O-H • -N-H • $>\text{C}=\text{O}$ • -C-O- • -C-H • $-\text{C}\equiv\text{N}$ • $-\text{NO}_2$ • Aromatic Substitutions <p>16.5.24 What is polymorphism and how does it influence IR spectra?</p> <p>16.5.25 Describe how to prepare the following:</p> <ul style="list-style-type: none"> • KBr pellet • Cast film • Sandwiched thin film 	

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<ul style="list-style-type: none"> Film deposited on KBr Nujol mull <p>16.5.26 How does over or under-grinding KBr/sample mixtures influence the IR spectra?</p> <p>16.5.27 What model IR does your laboratory use?</p> <p>16.5.27.1 What radiation sources and detectors are used in the FTIR and its attachments in your laboratory?</p> <p>16.5.28 What problems are encountered in using IR as a quantitative technique?</p> <p>16.5.29 What causes a sloped baseline?</p> <p>16.5.30 Explain baseline correction and how it is performed.</p> <p>16.5.31 What is spectral subtraction and under what conditions is it possible?</p> <p>16.5.32 What are the differences between background subtraction and spectral subtraction?</p> <p>16.5.33 What is the relationship between resolution and data point spacing?</p> <p>16.5.34 What resolution are samples normally run in your laboratory?</p> <p>16.5.35 What computer libraries are available in your lab and what are the resolutions of the spectra contained in them?</p> <p>16.5.36 Describe how a spectrum is auto-saved and/or saved.</p> <p>16.5.37 Describe how ATR analysis can be run on powders, liquids and mixtures.</p> <p>16.5.38 Explain any macros used in your lab and how they work.</p> <p>16.5.39 What are the advantages/disadvantages of a GC/MS compared to an IR when used for identification purposes?</p> <p>16.5.40 Describe the preventative maintenance schedule and the QA/QC procedures performed on the IR including the VAL-Q software.</p> <p>16.5.41 Describe as to a jury how an FTIR operates.</p> <p>16.6 Practical Exercises</p> <p>16.6.1 Using the standards in the laboratory, prepare the following samples, analyze via FTIR, and discuss the differences in the spectra:</p> <ul style="list-style-type: none"> Methamphetamine HCl, Phentermine HCl, Ephedrine HCl (ATR) Cocaine base, Cocaine HCl (KBr pellets and ATR) MDA, MDMA, MDEA (KBr pellets or ATR) <p>16.6.2 Obtain a mixture from the TC. Using spectral subtraction determine the two components present. Devise and carry out a physical separation of the two components and verify with FTIR.</p> <p>16.6.3 When available, run standards of methamphetamine and phentermine on the GC/FTIR.</p> <p>16.6.4 Obtain samples from the TC including procaine HCl, cocaine base/procaine mixture, Amoxicillin and gamma-butyrolactone and run using the ATR.</p>	

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<div>16.7 Modes of Evaluation</div> <div> <div>16.7.1 Written examination</div> <div>16.7.2 Court exercise (mini-mock trial)</div> </div> <div>◆ End</div>	